PB-245 598

PRELIMINARY MILE REPORT

Arthur N. Jarvis, et al

National Environmental Research Center
Las Vegas, Nevada

June 1975

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE

289058

EPA-680/4-75-007
JUNE 1975

PB 245 598

Environmental Monitoring Series

PRELIMINARY MILK REPORT

NATIONAL ENVIRONMENTAL RESEARCH CENTER
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89114

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
US Department of Commerce
Springfield, VA. 22151

RESEARCH REPORTING SERIES

Research reports of the Office of Research and Development, U. S. Environmental Protection Agency, have been grouped into five series. These five broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The five series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies

This report has been assigned to the ENVIRONMENTAL MONITORING series. This series describes research conducted to develop new or improved methods and instrumentation for the identification and quantification of environmental pollutants at the lowest conceivable significant concentrations. It also includes studies to determine the ambient concentrations of pollutants in the environment and/or the variance of pollutants as a function of time or meteorological factors.

EPA REVIEW NOTICE

This report has been reviewed by the National Environmental Research Center-Las Vegas, EPA, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the U.S. Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation of use.

Document is available to the public for sale through the National Technical Information Service, Springfield, Virginia 22161.

ia

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-680/4-75-007		

4. TITLE AND SUBTITLE	5. REPORT DATE
Preliminary Milk Report	June 1975
	6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
Arthur N. Jarvis and David G. Easterly	

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
Technical Support Laboratory National Environmental Research Center P. O. Box 15027 Las Vegas, Nevada 89114	1HA327
	11. CONTRACT/GRANT NO. In-house report

12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
Office of Research and Development U.S. Environmental Protection Agency Washington, D.C. 20460	14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

A summary of the results of the milk cross-check program from July 1973 through November 1974 is given. Examination of these results reveals that 29% of the participants were within the plus or minus 3 sigma control limits for strontium-89 analysis, 42% for the strontium-90, 52% for barium-140, 60% for iodine-131, and 77% for cesium-137. These results indicate the need for improvement in analytical procedures for the radionuclides studies. Accurate analysis of strontium-89 was particularly difficult for most laboratories to achieve, while the accurate analysis of cesium-137 was the least difficult.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
milk analysis isotopes ^{89}Sr, ^{90}Sr, ^{140}Ba, ^{131}I, ^{137}Cs quality control quantitative analysis laboratory performance	isotopes quality assurance radioactivity radiochemistry	07 05 12 02 14 07 18 02, 04, 08

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
Release unlimited	unclassified	43
	20. SECURITY CLASS *(This page)*	22. PRICE
	unclassified	3.75

EPA Form 2220-1 (9-73)

GPO 693-740/60

EPA-680/4-75-007
June 1975

PRELIMINARY MILK REPORT

by

Arthur N. Jarvis and David G. Easterly
Quality Assurance Branch
Technical Support Laboratory
National Environmental Research Center
Las Vegas, Nevada

ROAP 21BEK/22AAJ
Program Element 1HA327

NATIONAL ENVIRONMENTAL RESEARCH CENTER
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89114

ABSTRACT

A summary of the results of the milk cross-check program from July 1973 through November 1974 is given. These data indicate a need for improvement in the laboratory analysis of both beta and gamma-emitting radionuclides in milk. Also, the expected relationship, i.e., with increasing sample activity the accuracy of the analysis should improve, was not seen with any consistency at the levels of activity used in these studies. Accurate analysis of strontium-89 was particularly difficult for most laboratories to achieve, while the accurate analysis of cesium-137 was the least difficult.

These results exemplify the need for improvement in analytical procedures through the adoption of standard reference methods, more frequent calibration and maintenance of counting instruments, improved quality control practices within each laboratory, and greater participation in laboratory intercomparison studies conducted by the National Bureau of Standards, the Energy Research and Development Administration, and the U.S. Environmental Protection Agency.

CONTENTS

	Page
Abstract	iii
List of Tables and Figures	v
Introduction	1
Methods and Procedures	2
Results and Discussion	3
Summary	6
References	31
APPENDIX. Statistical Calculations	32

TABLES

Number		Page
1	Summary of Cross-Check Programs	8
2	Summary of Milk Analysis Data, July 1973 to November 1974	9
3	Limits Established by the Analytical Quality Control Service	12
4	Results of Milk Cross-Check Analyses, July 1973 to November 1974	12

FIGURES

Number		Page
1	Sample Analysis and Report of Participant's Data	13
2	Control Chart	15
3-A	Histogram of Monthly Analysis for Strontium-89	17
3-B	Histogram of Monthly Analysis for Strontium-90	20
3-C	Histogram of Monthly Analysis for Barium-140	23
3-D	Histogram of Monthly Analysis for Iodine-131	25
3-E	Histogram of Monthly Analysis for Cesium-137	28

PRELIMINARY MILK REPORT

INTRODUCTION

Environmental radiation measurements are made daily by federal, state, local, and private agencies. The data obtained from these measurements are utilized by the U.S. Environmental Protection Agency (EPA) and other agencies for such purposes as estimating dose and health effects, establishing standards and guides, and conducting regulatory activities. It is therefore imperative that the precision and accuracy of the data be assured so that policy decisions concerning environmental quality are based on valid and comparable data.

The present radiation quality assurance program of the EPA is designed to encourage the development and implementation of quality control procedures at all levels of sample collection, analysis, data processing, and reporting. As an integral part of the EPA's program, the Quality Assurance Branch of the National Environmental Research Center-Las Vegas (NERC-LV) distributes calibrated radionuclide solutions for instrument calibration and chemical yield determinations, and conducts a number of laboratory performance studies involving the analysis of radionuclides in environmental media.

The intercomparison studies program enables participating laboratories to maintain checks on their analyses and assists them in documenting the validity of their data. In addition, this program enables the EPA to obtain an overall estimate of the precision and accuracy of environmental radiation measurements, or more precisely the precision and accuracy of laboratory radioassay procedures of environmental samples.

Studies currently in progress involve samples of most environmental media and include milk, air, water, soil, diet, urine, and noble gases. Table 1 is a summary of the cross-check programs. Participants include nuclear facilities or their contractors, state, federal, and international laboratories. The number of participants has increased steadily during the past two years. Because of the large number of participants and the continuing nature of the programs, sufficient data are generated to enable valid periodic statistical assessment of the quality of environmental data.

Laboratories perform the analyses and return their data to the Quality Assurance Branch for statistical analysis. Comparisons are made between laboratories and within an individual laboratory for accuracy and precision. A computer report and a periodically updated performance chart are returned to each participant. This enables each laboratory to document the precision and accuracy of its radiation data, to identify instrumental and procedural problems, and to compare performance with other laboratories.

METHODS AND PROCEDURES

Through December 1973, reconstituted powdered milk was used in the milk cross-check program. Two modifications were made to the program beginning in 1974: (1) fresh, whole, pasteurized, homogenized milk was used in preparing the samples, and (2) sample distribution was changed from monthly to a bimonthly basis.

The milk was prepared for distribution by thoroughly mixing with 10 ml/gallon of formalin and 3.79 ml/gallon of antifoaming agent (Dow Corning Antifoam,^{R} FG-10 Emulsion, Food Grade). Three aliquots were gamma counted for background determination. The remaining milk was "spiked" using calibrated solutions of the radionuclides. The milk was again thoroughly mixed and three aliquots taken for gamma analysis. If these results indicated homogeneity, the milk was packaged for

shipment. A 4-liter sample containing strontium-89, strontium-90, cesium-137, iodine-131, and barium-140 was distributed to each of approximately 35 participating laboratories.

Each participating laboratory made three independent determinations for each radionuclide included in the particular cross-check sample and reported its results to the Quality Assurance Branch. Control limits (sigma limits) previously established by the Analytical Quality Control Service in Winchester, Massachusetts, were used in analyzing the quality of the results obtained by these laboratories (1,2). These limits were based on the purpose for which the data were being obtained and on reasonable laboratory ability. Upon receipt of the reports from all participating laboratories, the data were transferred to punch cards and analyzed using a CDC 6400 computer. This analysis included determination of the experimental average and standard deviation of the sample, the normalized range, standard error normalized deviation, experimental sigma, and the grand average of all laboratories for each radionuclide. Examples of sample calculations to illustrate the computations performed by the computer are shown in the appendix.

A report was generated containing the data reported by all participating laboratories, listed according to their identity code, along with the results of the data analysis. Examples are shown in Figure 1. In addition, a control chart was generated for each radionuclide included in the sample (Figure 2). The control charts are updated each time a laboratory participates in a cross-check study, thus giving each laboratory a continuous record of its performance.

A copy of the computer printout and a control chart for each radionuclide is mailed to each participant approximately two weeks following the report due date.

RESULTS AND DISCUSSION

A summary of the results of the milk analysis data is shown in Table 2. All participants received samples containing strontium-89,

strontium-90, cesium-137, iodine-131, and barium-140; however, all did not submit results for each analysis. This resulted in a different number, N, being reported for the various radionuclides.

A comparison may be made between the experimental standard deviation, s, calculated from the data supplied by the participating laboratories (Table 2) and the standard deviation, σ, expected for various analyses listed in Table 3.

For the purpose of comparison, the experimental [(s/known)100] and expected [(σ/known)100] standard deviations, expressed as a percent of the known values, are shown for each month in Table 2. The experimental standard deviation divided by the known value (s/μ) indicates no improvement in the analytical results when whole milk was used instead of powdered.

Figure 3-A shows the results in the form of a histogram for the strontium-89 analysis by month.* The February results were the poorest over the period for which strontium-89 data were analyzed. When comparing the grand average ($\bar{X}$) to the known value (μ), a negative bias was observed for all monthly samples except February. No explanation for either the negative bias or the poor analytical performance by the participating laboratories is readily apparent. It is now necessary to determine if this is due to the analytical method(s) being used and if so if a different method of analysis or modification of the existing method(s) is warranted.

The monthly results of the strontium-90 analysis are shown in Figure 3-B. On an average the (s/μ) values were lower and the deviations from μ were less than for strontium-89 analysis. Even though the results show some inconsistency from month to month, in general they may be viewed with some optimism since data for at least one

---

* For all data used in this report, Chauvenet's criterion was used as the basis for acceptance or rejection of the $\bar{X}$ value of each laboratory.

month are sufficiently accurate to indicate good results are attainable. More frequent calibration and maintenance of counting instruments, improved quality control practices within the laboratories, and greater participation in laboratory intercomparison studies may be beneficial for some of the laboratories having analytical difficulties.

The monthly results of the barium-140 analysis are shown in Figure 3-C. Because of the manufacturer's production schedules and the half-life of barium-140, some monthly cross-check samples did not contain this isotope. Unlike strontium-89 and 90, generally no "wet" chemistry is involved in the barium-140 analysis. Most analysts determine the gamma emitters by gamma spectroscopy. This would indicate errors arise from improper, infrequent, or erroneous calibration; faulty or inadequate instrumentation; or erroneous processing and interpretation of data. Participants obtaining inaccurate results for their barium analysis should restandardize and calibrate their instruments more frequently using the same geometry as that used for sample counting. It appears from this limited amount of data that the barium-140 results are better in terms of accuracy and (s/μ) than the strontium analysis.

The monthly results of the iodine-131 analysis are shown in Figure 3-D. These results are as good as the barium-140 results in terms of a small deviation from μ, and are better than the barium-140 results in terms of s/μ. With proper calibration and interpretation, accurate results appear readily attainable. During the six-month period where reconstituted powdered milk was used, the results were relatively good, with two of the six analyses having an experimental sigma value of less than 10 pCi/liter. Results were the best for the September sample.

The monthly results of the cesium-137 analysis are shown in Figure 3-E. These results are considerably better in terms of the ratio s/μ than those for any of the other radionuclides used in the milk cross-check program. The reason for the better results with cesium may be partially attributed to the fact that many participants standardize their instruments with a cesium-137 source. The cesium-137 peak area

may also be somewhat easier defined than the iodine-131 and barium-140 peaks. Even better results for cesium-137 analysis could be attained if improvement was made by a few of the participants. This may best be accomplished through consultation with the laboratories having the analytical problem.

Table 3 gives the limits previously established by the Analytical Quality Control Service (1,2). During the period July 1973 to November 1974, with few exceptions, results from the milk cross-check program did not approach these established limits. Examination of these results reveals that 29% of the participants were within the plus or minus 3 sigma control limits for the strontium-89 analysis, 42% for the strontium-90, 52% for barium-140, 60% for iodine-131, and 77% for cesium-137 (Table 4).

SUMMARY

Data compiled for the milk cross-check program from July 1973 to November 1974 indicate the need for improvement in analytical procedures for all radionuclides studied. Accurate analysis of strontium-89 was particularly difficult for most laboratories to achieve, while the accurate analysis of cesium-137 was the least difficult.

It is hoped that results similar to those obtained for cesium-137 can be accomplished for analysis of the other radionuclides. The data, although generally having relatively large measures of random deviations, indicate this may be possible for all except strontium-89. There was at least one month where the experimental sigma approached the established limits for all radionuclides except strontium-89. In September 1973 the cesium-137 experimental sigma was less than the established limits. These data would indicate the laboratories can achieve good accuracy for most of the samples being analyzed.

Two things are apparent from these data. The expected relationship, i.e., the accuracy of the analysis should improve with increasing sample activity, was not seen with any consistency at the levels

of activity used in the studies (Table 2). There is a need for considerable improvement in the laboratory analysis of both beta and gamma-emitting radionuclides in milk.

One reason for the variability in the results may be due to the expansion of the milk cross-check program to include new participants. The number of participants more than doubled during the first half of 1973. As these laboratories become more familiar with the analysis, hopefully the accuracy of the results will improve. For strontium-89, and possibly strontium-90, it appears that new methods of analysis should be evaluated since less than a third of the participating laboratories are doing a satisfactory job of strontium-89 analysis and less than half for strontium-90. If data over the next several months indicate that the present limits are unrealistic for some radionuclides and cannot be attained by the majority of participants, then the limits should be made more realistic. Available information does indicate good internal precision for most laboratories, but poor accuracy in many cases. This exemplifies the need for improvement in analytical procedures through the adoption of standard reference methods and protocols for establishing equivalency to the reference method, more frequent calibration and maintenance of counting instruments, improved quality control practices within each laboratory, and greater participation in laboratory intercomparison studies conducted by the National Bureau of Standards, the Energy Research and Development Administration, and the Environmental Protection Agency.

Table 1. SUMMARY OF CROSS-CHECK PROGRAMS*

SAMPLE	ANALYSIS	ACTIVITY PER ISOTOPE	QUANTITY SUPPLIED	PRESERVATIVE	DISTRIBUTION	TIME FOR ANALYSIS & REPORT
Milk	^{89}Sr, ^{90}Sr, ^{131}I, ^{137}Cs, ^{140}Ba, K	< 200 pCi/l	~ 4 liters	Formalin	Bimonthly	6 weeks
Water						
Gross α, β*	Gross α, β	< 100 pCi/l	~ 4 liters	0.5 N HNO_3	Bimonthly	4 weeks
Gamma	^{60}Co, ^{106}Ru, ^{134}Cs, ^{137}Cs, ^{51}Cr, ^{65}Zn	< 500 pCi/l	~ 4 liters	0.5 N HNO_3	Bimonthly	4 weeks
^{3}H	^{3}H	< 3500 pCi/l	~ 60 ml	none	Bimonthly	4 weeks
^{239}Pu*	^{239}Pu	< 10 pCi/l	~ 4 liters	0.5 N HNO_3	Semiannually	8 weeks
^{226}Ra	^{226}Ra	< 20 pCi/l	~ 4 liters	0.5 N HNO_3	Quarterly	6 weeks
Air						
Gross α, β*	α, β, γ	< 200 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	4 weeks
^{239}Pu*	^{239}Pu	< 2 pCi/sample	3 - 2" or 4" diam. air filters	none	Quarterly	6 weeks
Soil*	^{239}Pu	< 50 pCi/sample	~ 100 g	none	Semiannually	8 weeks
Diet	^{89}Sr, ^{90}Sr, ^{131}I, ^{137}Cs, ^{140}Ba, K	< 200 pCi/kg	3 - 4-liter samples	Formalin	Quarterly	8 weeks
Urine	^{3}H	< 3500 pCi/l	~ 60 ml	Formalin	Quarterly	8 weeks
Gas	^{85}Kr	< 20 pCi/ml	10 liters	none	Semiannually	8 weeks

* Laboratories are required to have the necessary licenses before receiving these samples.

Table 2. SUMMARY OF MILK ANALYSIS DATA, JULY 1973 TO NOVEMBER 1974

	Jul'73	Aug'73	Sep'73	Oct'73	Nov'73	Dec'73	Feb'74	Apr'74	Jul'74	Sep'74	Nov'74
Strontium-89											
Known pCi/liter	98	96	85	133	123	89	83	204	151	157	172
1 σ limit pCi/liter	5	5	5	7	6	5	5	10	8	7.9	8.6
1 σ/known %	5.1	5.2	5.9	5.3	4.9	5.6	6.0	4.9	5.3	5.0	5.0
N	17	14	13	19	16	18	23	28	26	23	30
$\bar{X}$ pCi/liter	87.2	61.5	76.9	112.3	98.3	69.5	92.6	171.9	128.1	137.8	137.3
s pCi/liter	11.0	29.6	12.3	25.4	24.9	30.8	44.0	36.7	27.6	25.7	39.1
s/known %	11.2	30.8	14.5	19.1	20.2	34.6	53.0	18.0	18.3	16.4	22.7
Strontium-90											
Known pCi/liter	65	158	72	119	135	103	198	172	152	124	102
1 σ limit pCi/liter	3	8	4	6	7	5	10	9	8	6.2	5.1
1 σ/known %	4.6	5.1	5.6	5.0	5.2	4.9	5.1	5.2	5.3	5.0	5.0
N	23	17	16	23	22	22	21	31	29	28	33
$\bar{X}$ pCi/liter	64.0	130.4	67.6	106.1	115.2	93.1	182.7	169.9	152.7	124.1	94.1
s pCi/liter	8.3	44.5	6.4	19.9	26.9	16.4	20.0	22.9	28.8	15.6	15.2
s/known %	12.8	28.2	8.9	16.7	19.9	15.9	10.1	13.3	18.9	12.6	14.9

Table 2 (continued). SUMMARY OF MILK ANALYSIS DATA, JULY 1973 TO NOVEMBER 1974

	Jul'73	Aug'73	Sep'73	Oct'73	Nov'73	Dec'73	Feb'74	Apr'74	Jul'74	Sep'74	Nov'74
Barium-140											
Known pCi/liter	148	152	146	104	0	0	159	0	0	193	0
1 σ limit pCi/liter	7	8	7	5			8			9.6	
1 σ/known %	4.7	5.3	4.8	4.8			5.0			5.0	
N	27	21	22	32			35			33	
$\bar{X}$ pCi/liter	152.6	158.4	145.3	107.4			159.3			195.2	
s pCi/liter	11.0	24.9	27.9	19.2			11.9			21.9	
s/known %	7.4	16.4	19.1	18.5			7.5			11.3	
Iodine-131											
Known pCi/liter	115	144	123	493	104	177	150	150	142	188	126
1 σ limit pCi/liter	6	7	6	25	5	9	8	8	7	9.4	6.3
1 σ/known %	5.2	4.9	4.9	5.1	4.8	5.1	5.3	5.3	4.9	5.0	5.0
N	33	22	22	34	26	25	40	41	35	31	36
$\bar{X}$ pCi/liter	125.1	147.8	121.3	486.3	99.7	175.8	149.9	154.6	145.8	192.2	122.4
s pCi/liter	18.3	13.7	8.8	28.2	11.0	9.6	27.7	20.7	15.9	20.4	10.5
s/known %	15.9	9.5	7.2	5.7	10.6	5.4	18.5	13.8	11.2	10.9	8.3

Table 2 (continued). SUMMARY OF MILK ANALYSIS DATA, JULY 1973 TO NOVEMBER 1974

	Jul'73	Aug'73	Sep'73	Oct'73	Nov'73	Dec'73	Feb'74	Apr'74	Jul'74	Sep'74	Nov'74
Cesium-137											
Known pCi/liter	116	122	84	91	126	101	214	174	155	126	101
1 σ limit pCi/liter	6	6	5	5	7	5	11	9	8	6.3	5.1
1 σ/known %	5.2	4.9	6.0	5.5	5.6	5.0	5.1	5.2	5.2	5.0	5.0
N	34	22	22	35	28	30	38	42	37	35	38
$\bar{X}$ pCi/liter	116.5	120.1	81.6	88.7	123.1	100.1	208.9	171.1	157.3	132.2	102.8
s pCi/liter	6.6	7.7	3.9	8.5	9.3	9.4	12.7	16.1	10.0	7.6	5.6
s/known %	5.7	6.3	4.6	9.3	7.4	9.3	5.9	9.3	6.5	6.0	5.5

Table 3. LIMITS ESTABLISHED BY THE ANALYTICAL QUALITY CONTROL SERVICE

Radionuclide	1σ Limits for Single Determination	
	For Levels ≤100 pCi/liter (pCi/liter)	For Levels >100 pCi/liter (%)
^{131}I	5	5
^{137}Cs	5	5
^{140}Ba	5	5
^{89}Sr	5	5
	For Levels ≤30 pCi/liter	For Levels >30 pCi/liter (%)
^{90}Sr	1.5	1.5

Table 4. RESULTS OF MILK CROSS-CHECK ANALYSES, JULY 1973 TO NOVEMBER 1974

σ Limits	Radionuclide				
	^{89}Sr	^{90}Sr	^{140}Ba	^{131}I	^{137}Cs
	Laboratories Within σ Limits (%)				
≤1	11.2	19.4	25.3	29.7	39.0
≤2	20.7	31.6	42.9	49.9	63.1
≤3	28.5	42.4	52.0	59.7	76.7
>3	71.5	57.6	48.0	40.3	23.3

NERC-LV RADIONUCLIDES IN MILK CROSS-CHECK PROGRAM --- JUNE 1975

6/15/75 SAMPLE - A 137CS

KNOWN VALUE = 101 PCI/L
EXPECTED LABORATORY PRECISION (1S, 1 DETERMINATION) = 5.1 PCI/L

LAB	RESULT	EXPERIMENTAL SIGMA	RNG ANLY (R + SR)	AVERAGE	NORMALIZED DEVIATION (GRAND AVG)	(KNOWN)
D	98					
D	102					
D	102	2.3	.46	101	-.7	-.1
P	NO DATA PROVIDED					
Q	104					
Q	104					
Q	108	2.3	.46	105	.9	1.5
R	95					
R	99					
R	93	3.1	.69	96	-2.4	-1.8
S	108					
S	105					
S	106	1.5	.35	106	1.2	1.8
V	96					
V	110					
V	98	7.6	2.18	101	-.5	.1

EXPERIMENTAL SIGMA (ALL LABS) = 7 GRAND AVERAGE = 103

* - NOT USED FOR CALCULATING GRAND AVERAGE
** - INSUFFICIENT INFORMATION TO CALCULATE

Figure 1. Sample Analysis and Report of Participant's Data

Explanation of terms in Figure 1:

Title:	Program name, sample collection date, sample code letter, analysis type, known concentrations of radionuclide, expected standard deviation of analysis - single determination.
Column 1:	Laboratory identification code (A, B, C, etc.).
Colume 2:	Laboratory results (0-25 results listed down column).
Column 3:	1s (standard deviation) of the experimental results.
Columns 4 and 5:	Normalized range value in "mean range + standard error of the range" ($\bar{R} + \sigma_R$ units for comparability. (See *Statistical Techniques for Quality Control of Environmental Radioassay*, AQCS Report Stat-1, November 1964, pages 4-8.) ($S_R = \sigma_R$ for printing purposes.)
Column 6:	Average value.
Column 7:	Normalized deviation from the grand average value of all laboratories expressed in σ_M units.
Column 8:	Normalized deviation from the known value expressed in σ_M units.
Bottom of Chart:	1s experimental error of all laboratories, and the grand average of all laboratories.

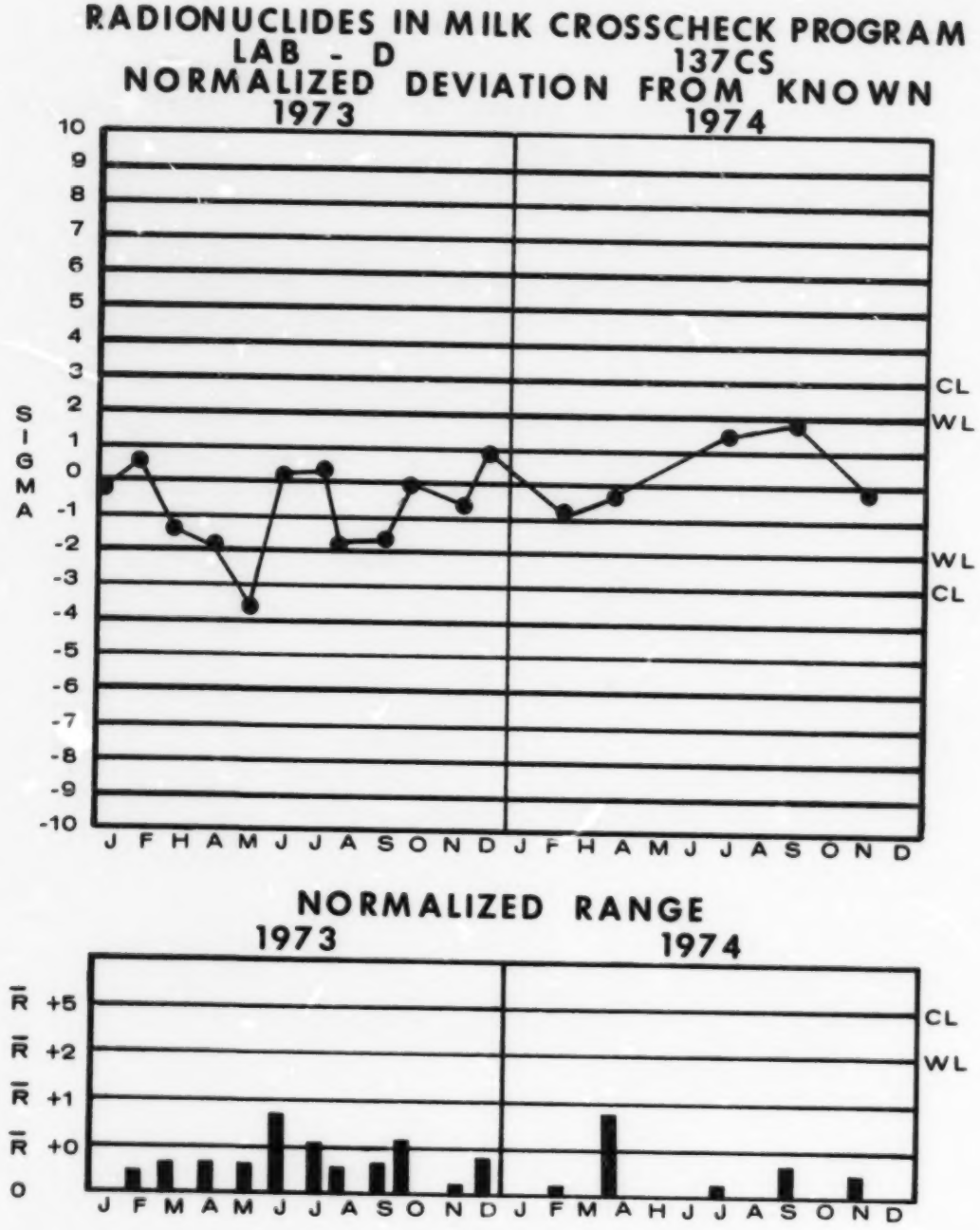


Figure 2. Control Chart

Explanation of terms used in the control chart (Figure 2).

Title:	Name of program, laboratory code letter, and type of analysis.
Upper Graph:	"Normalized deviation from known versus the month of analysis." [The 95.0% ($\mu \pm 2$ $_M$) and the 99.7% ($\mu \pm 3\sigma_M$) confidence levels were chosen as the warning levels and control limits respectively.]
Lower Graph:	"Normalized range values ($\bar{R} + \sigma_R$) versus the month of analysis." [The 97.5% ($\bar{R} + 2\sigma_R$) and ~100% ($\bar{R} + 3\sigma_R$) confidence levels were chosen as the warning levels and control limits respectively.]

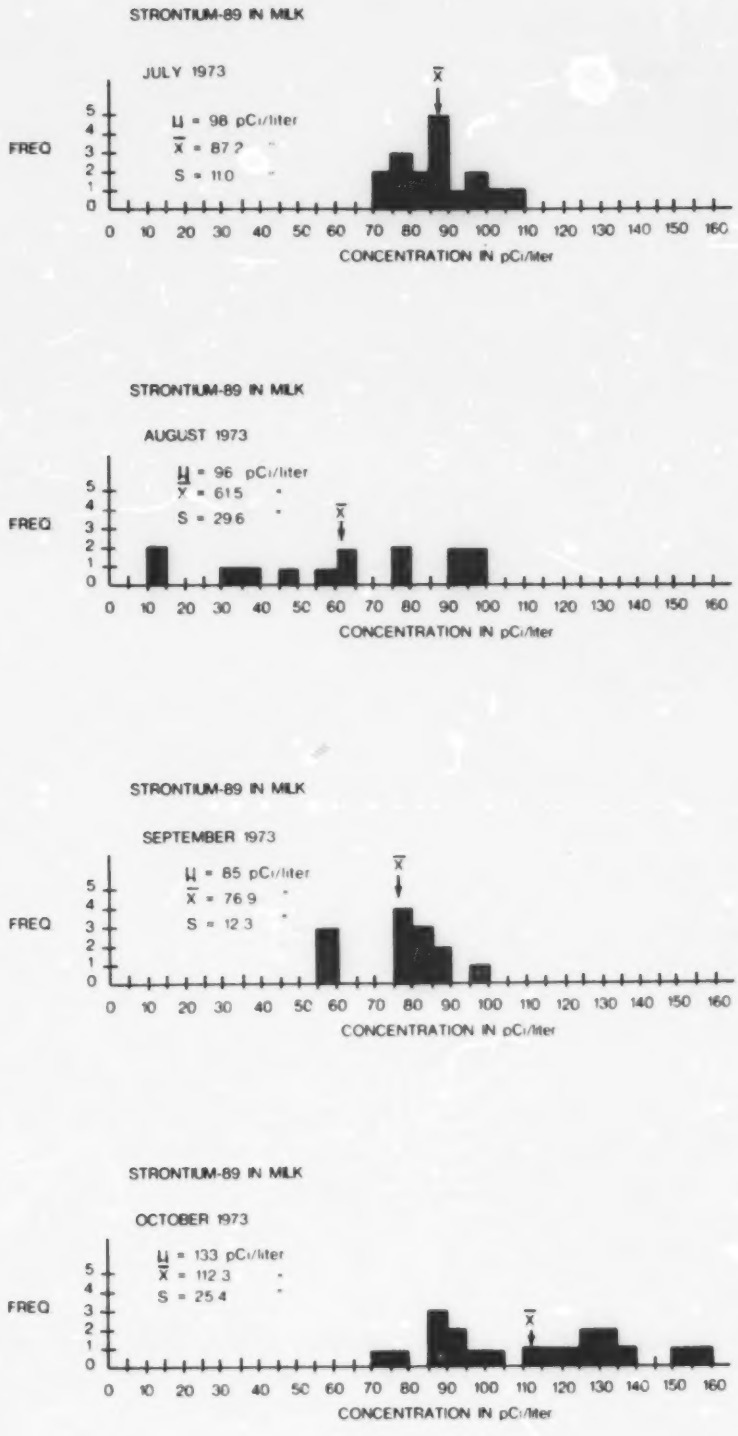
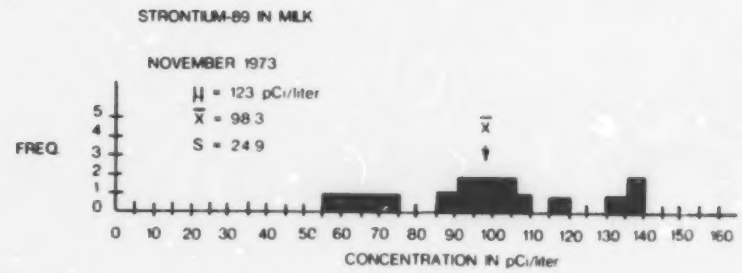
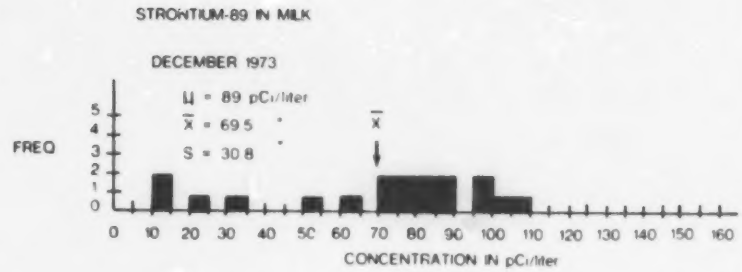
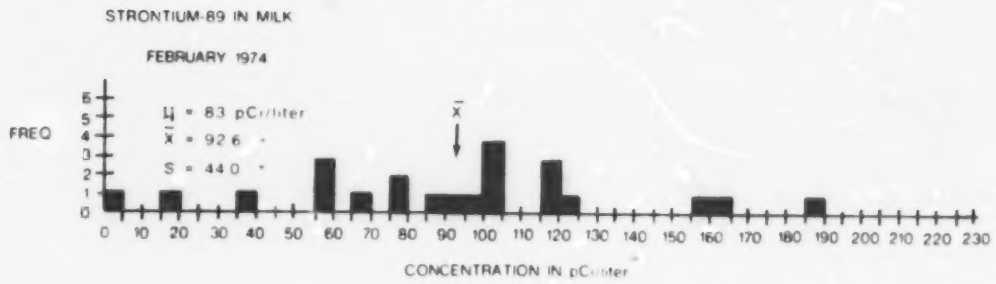


Figure 3-A. Histogram of Monthly Analysis for Strontium-89







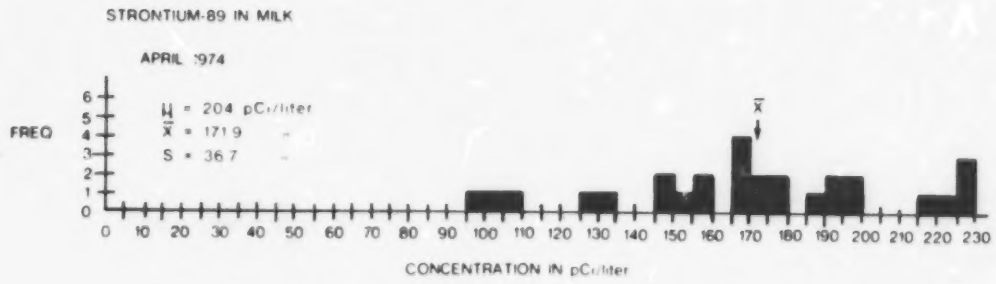
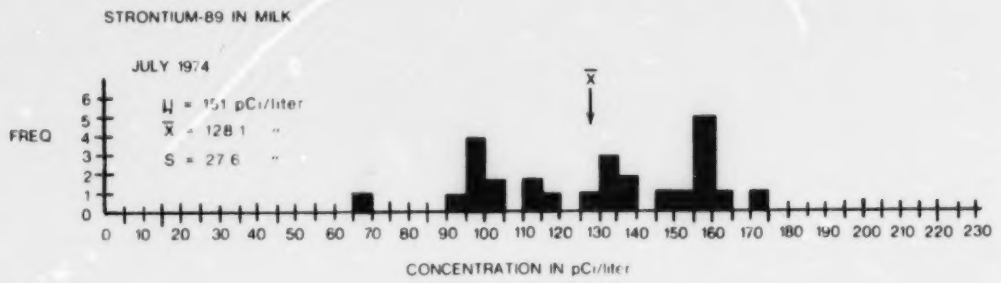
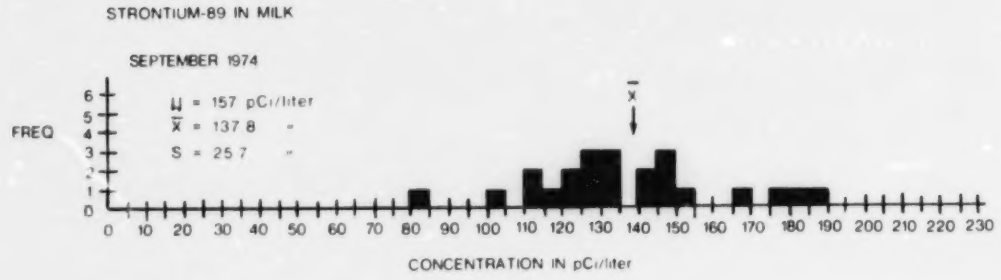


Figure 3-A. Histogram of Monthly Analysis for Strontium-89 (continued).





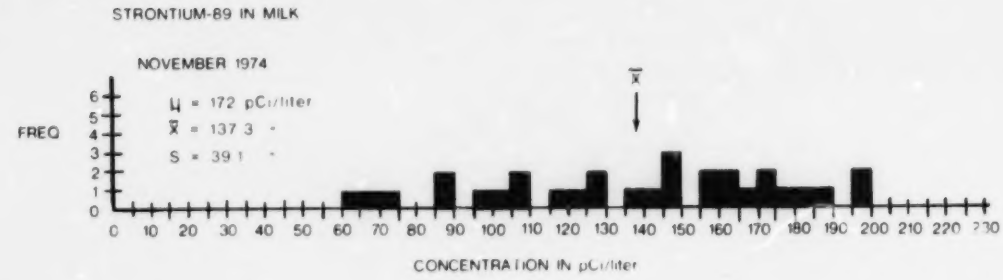


Figure 3-A. Histogram of Monthly Analysis for Strontium-89 (continued).

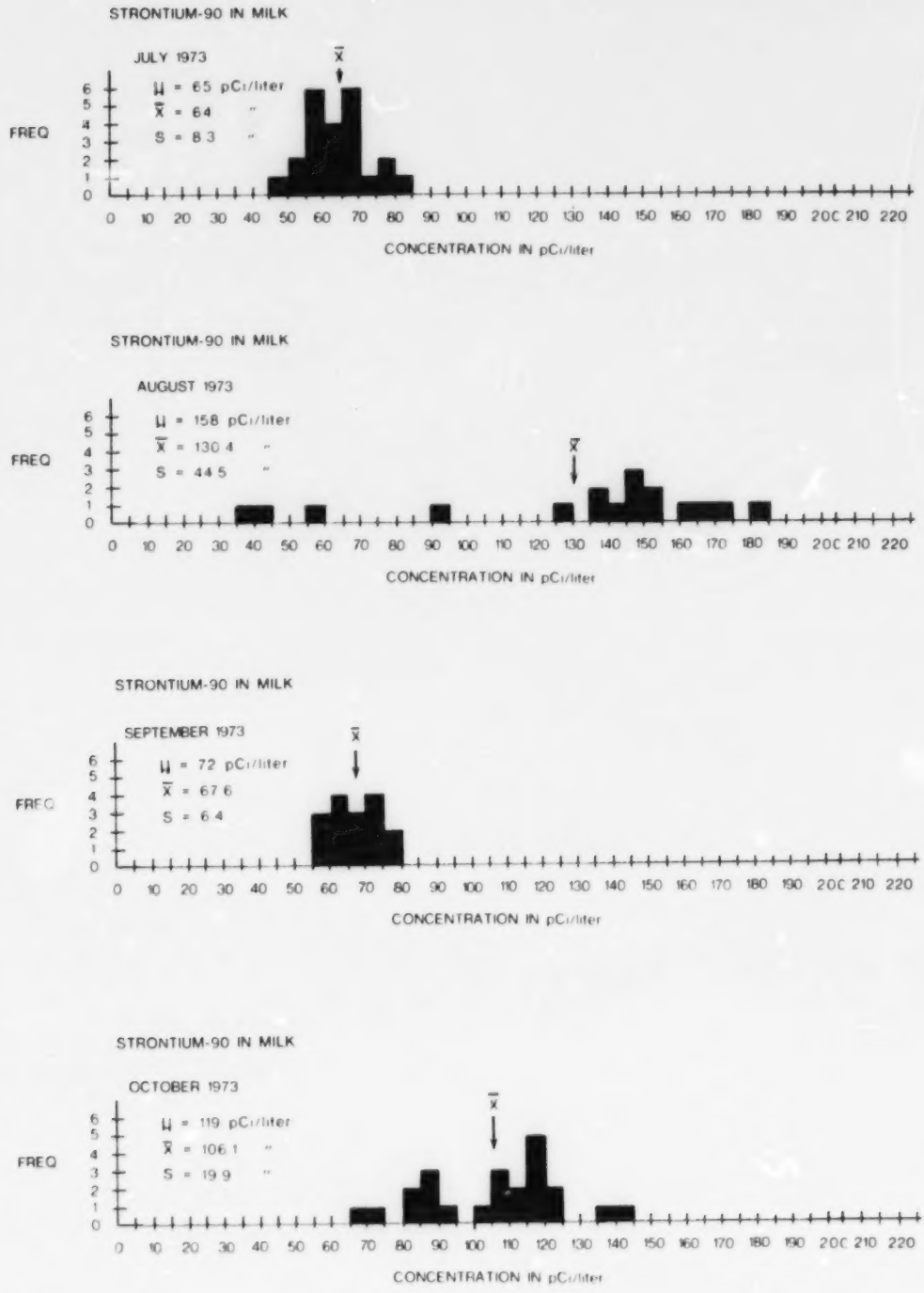


Figure 3-B. Histogram of Monthly Analysis for Strontium-90

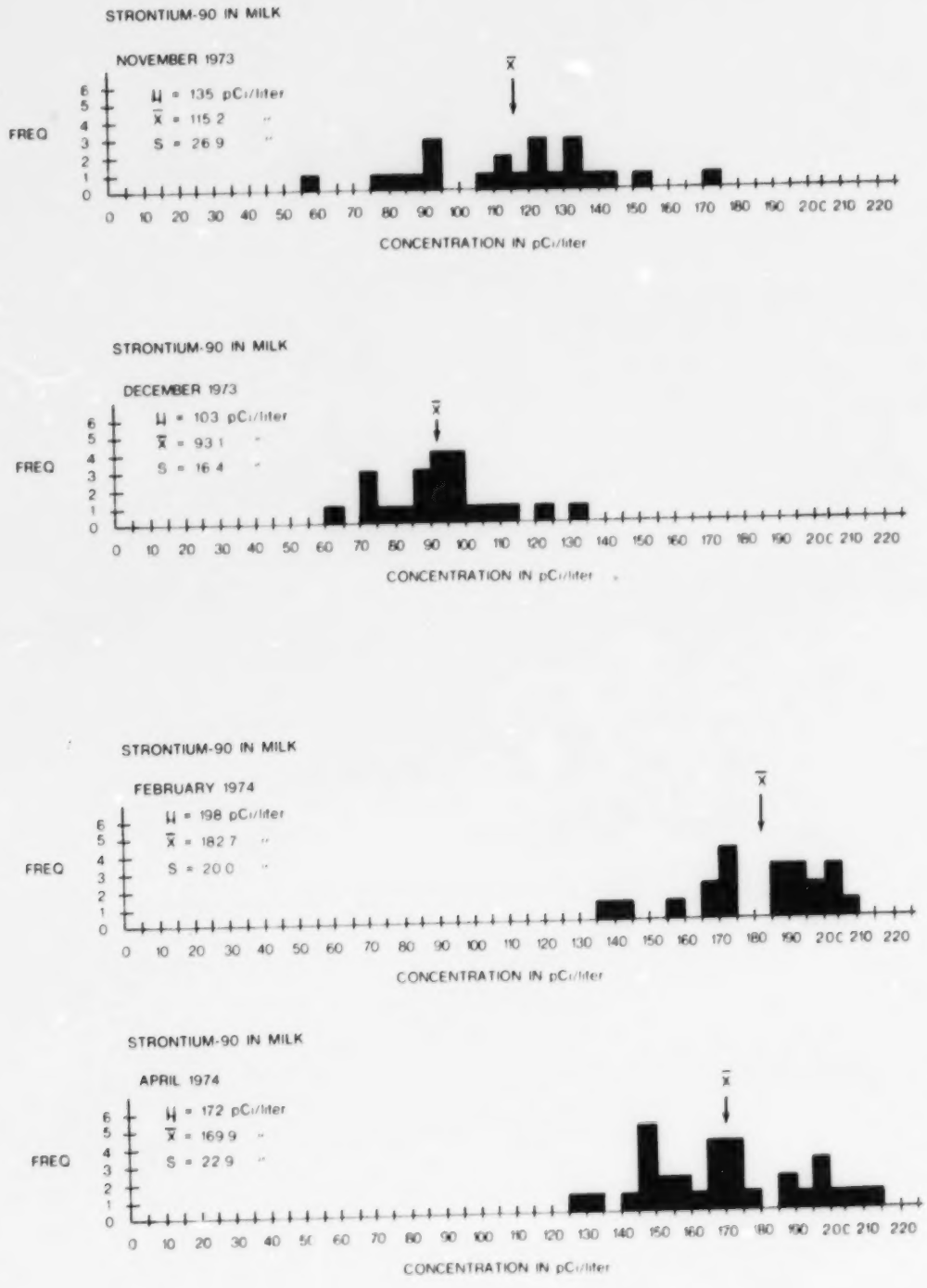


Figure 3-B. Histogram of Monthly Analysis for Strontium-90 (continued).

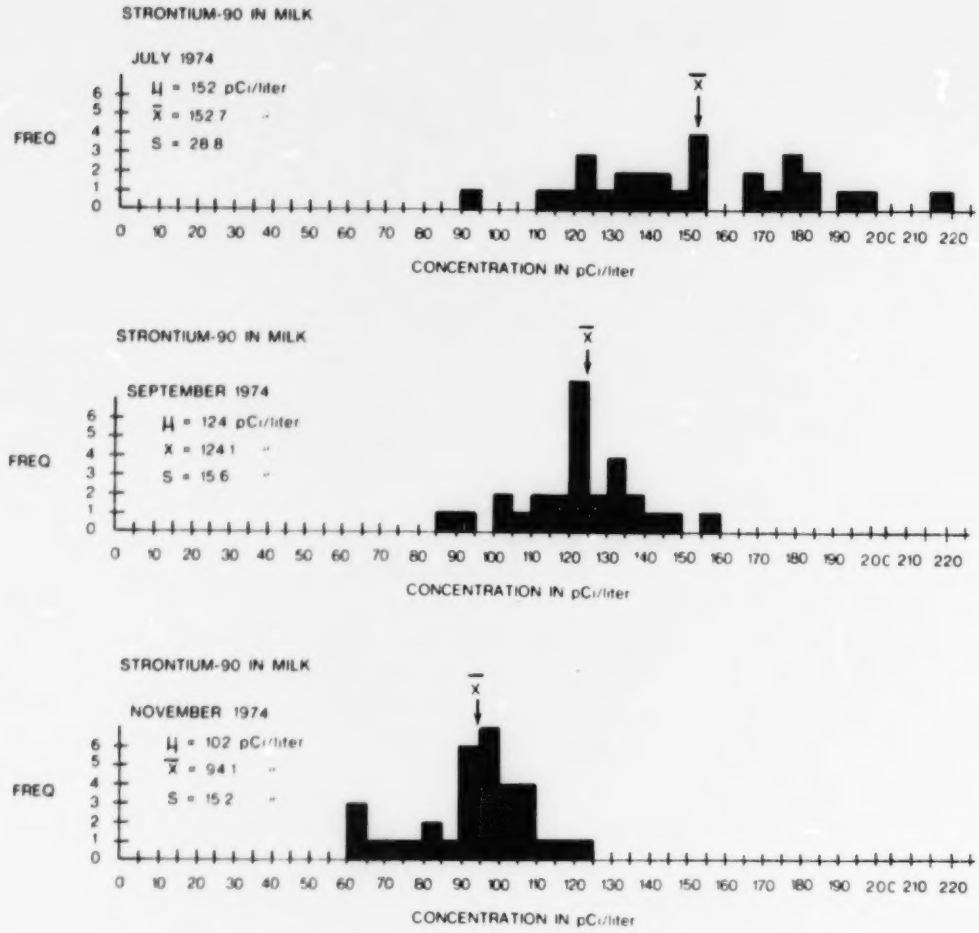


Figure 3-B. Histogram of Monthly Analysis for Strontium-90 (continued).

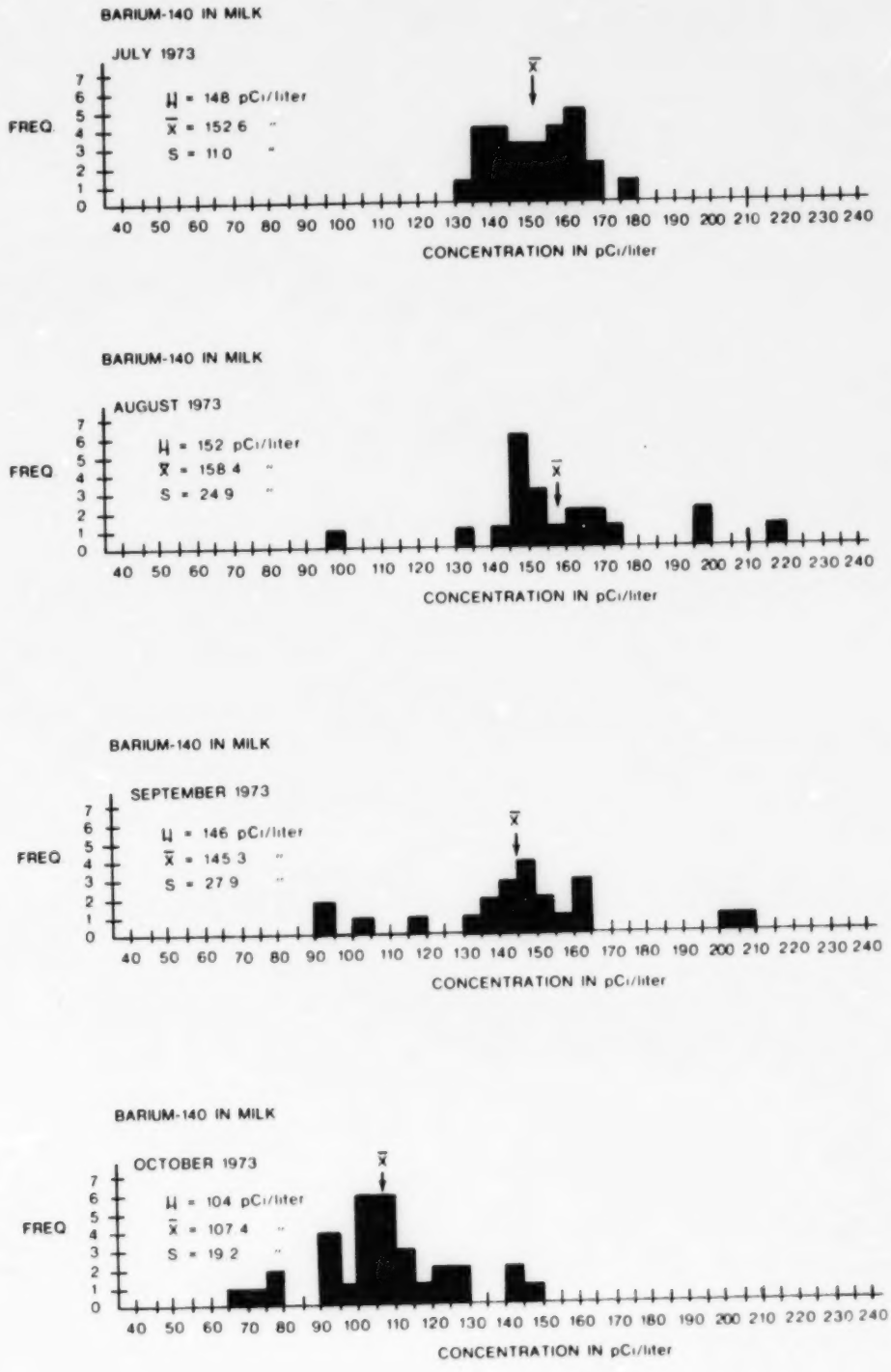
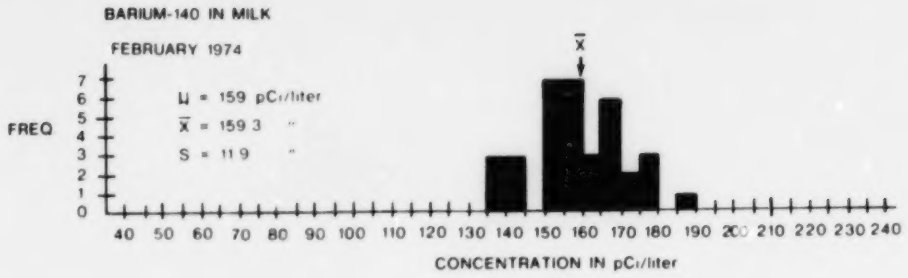


Figure 3-C. Histogram of Monthly Analysis for Barium-140



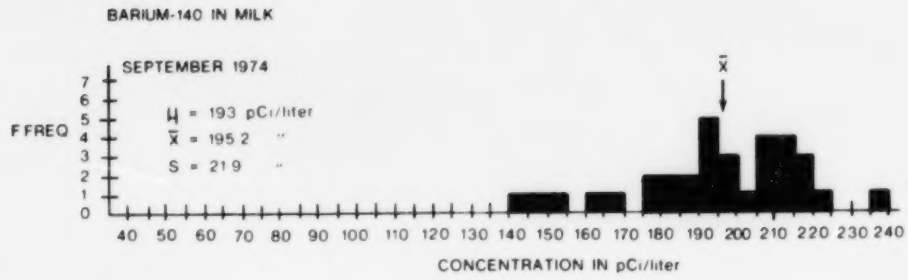


Figure 3-C. Histogram of Monthly Analysis for Barium-140 (continued).

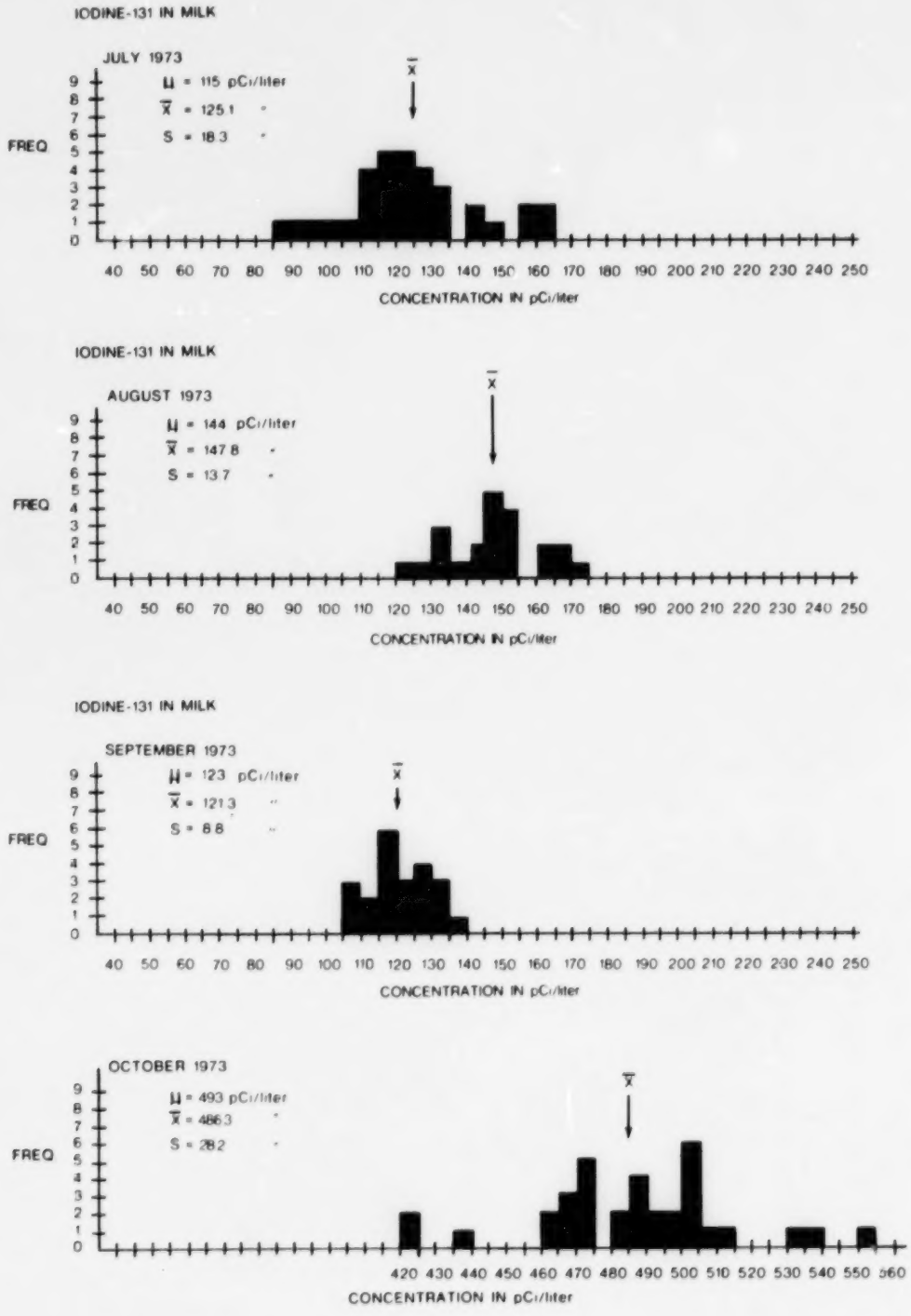


Figure 3-D. Histogram of Monthly Analysis for Iodine-131

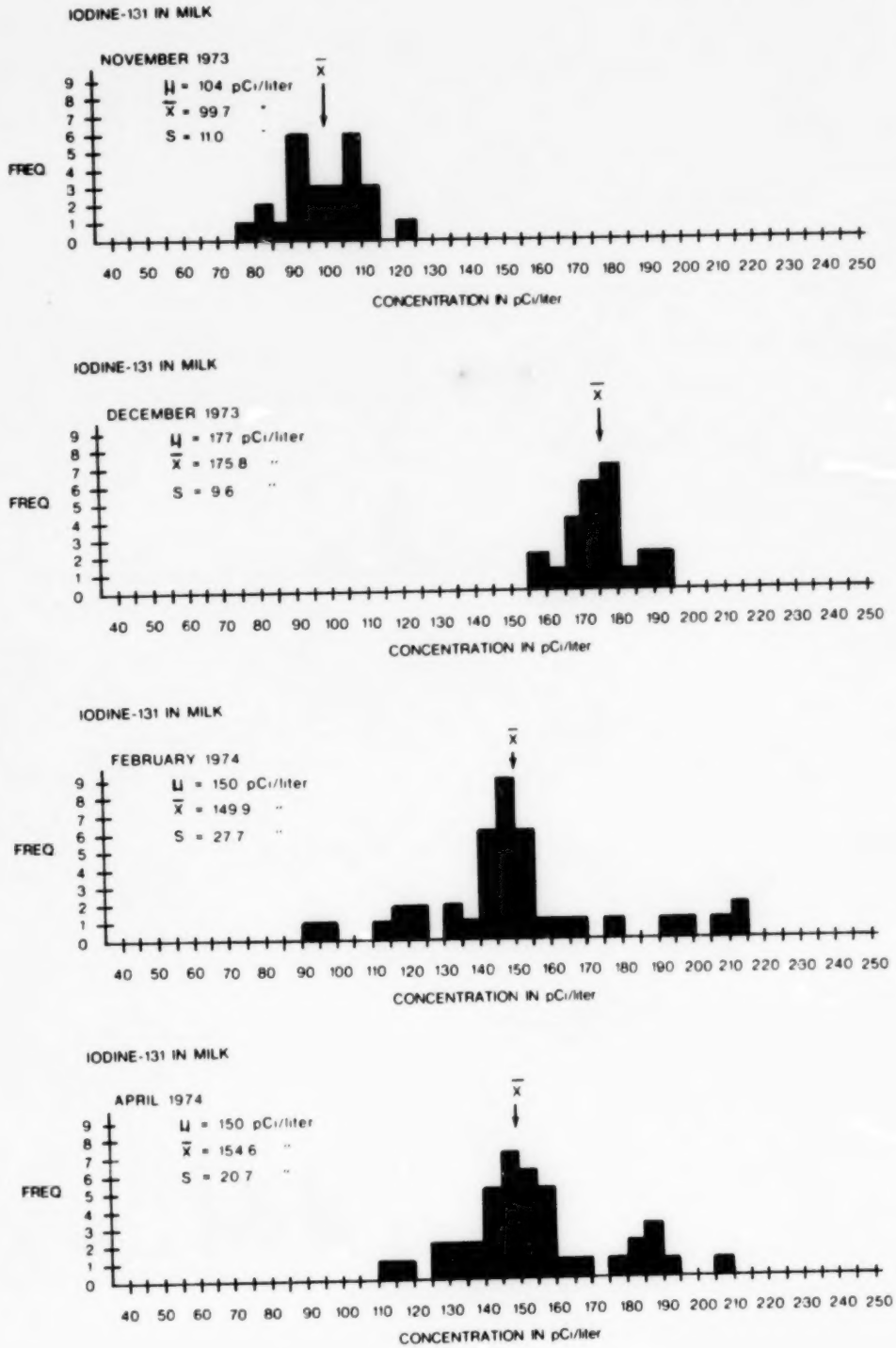


Figure 3-D. Histogram of Monthly Analysis for Iodine-131 (continued).

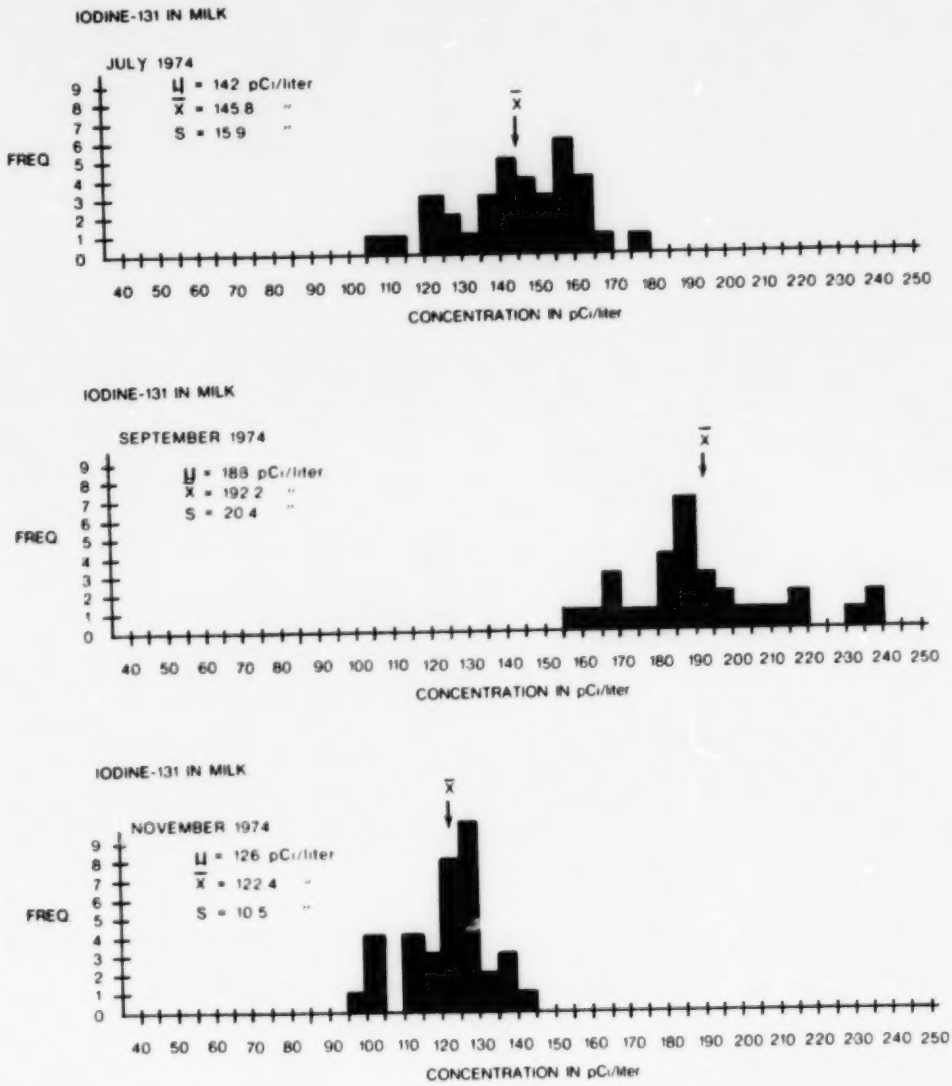


Figure 3-D. Histogram of Monthly Analysis for Iodine-131 (continued).

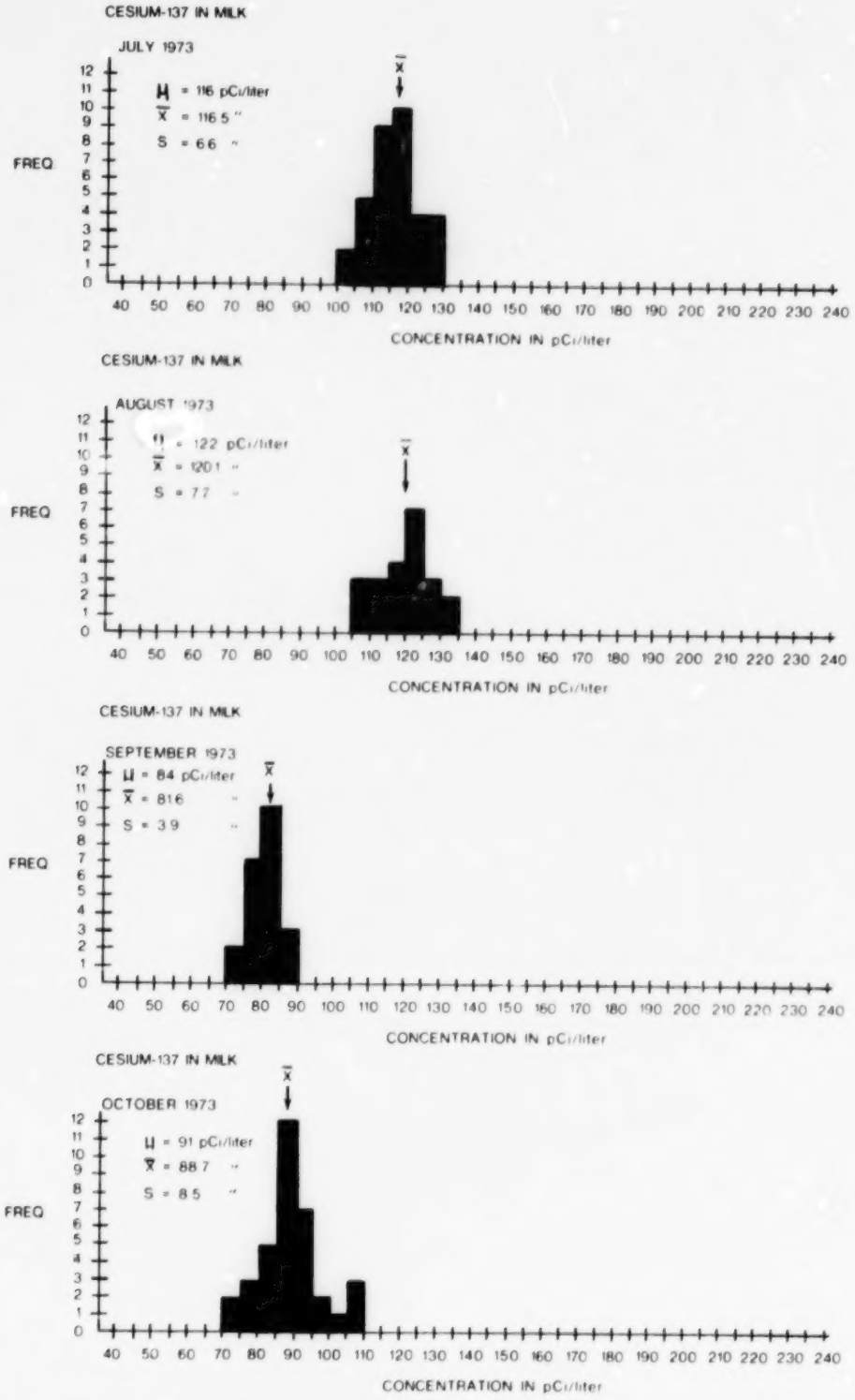


Figure 3-E. Histogram of Monthly Analysis for Cesium-137

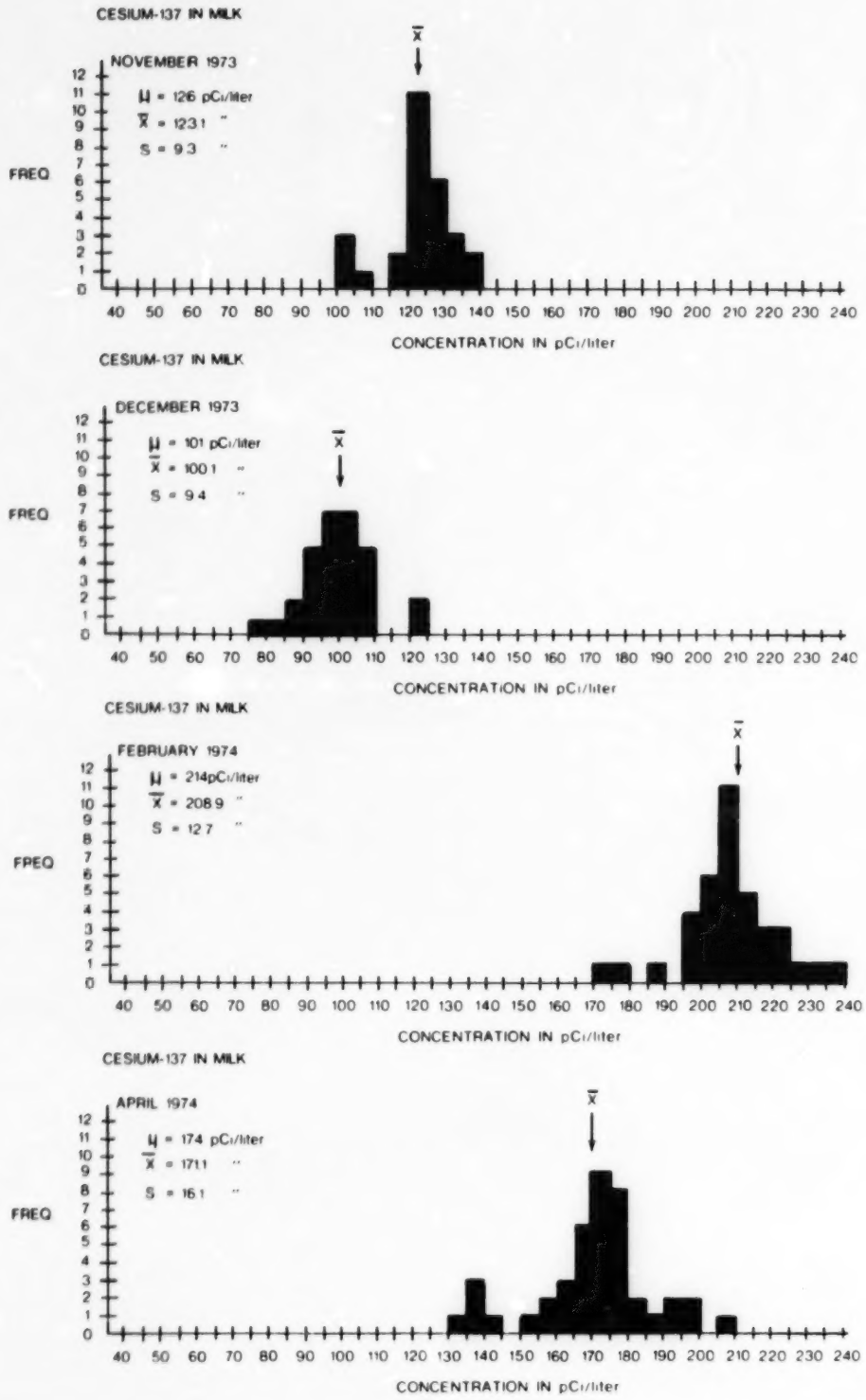


Figure 3-E. Histogram of Monthly Analysis for Cesium-137 (continued).

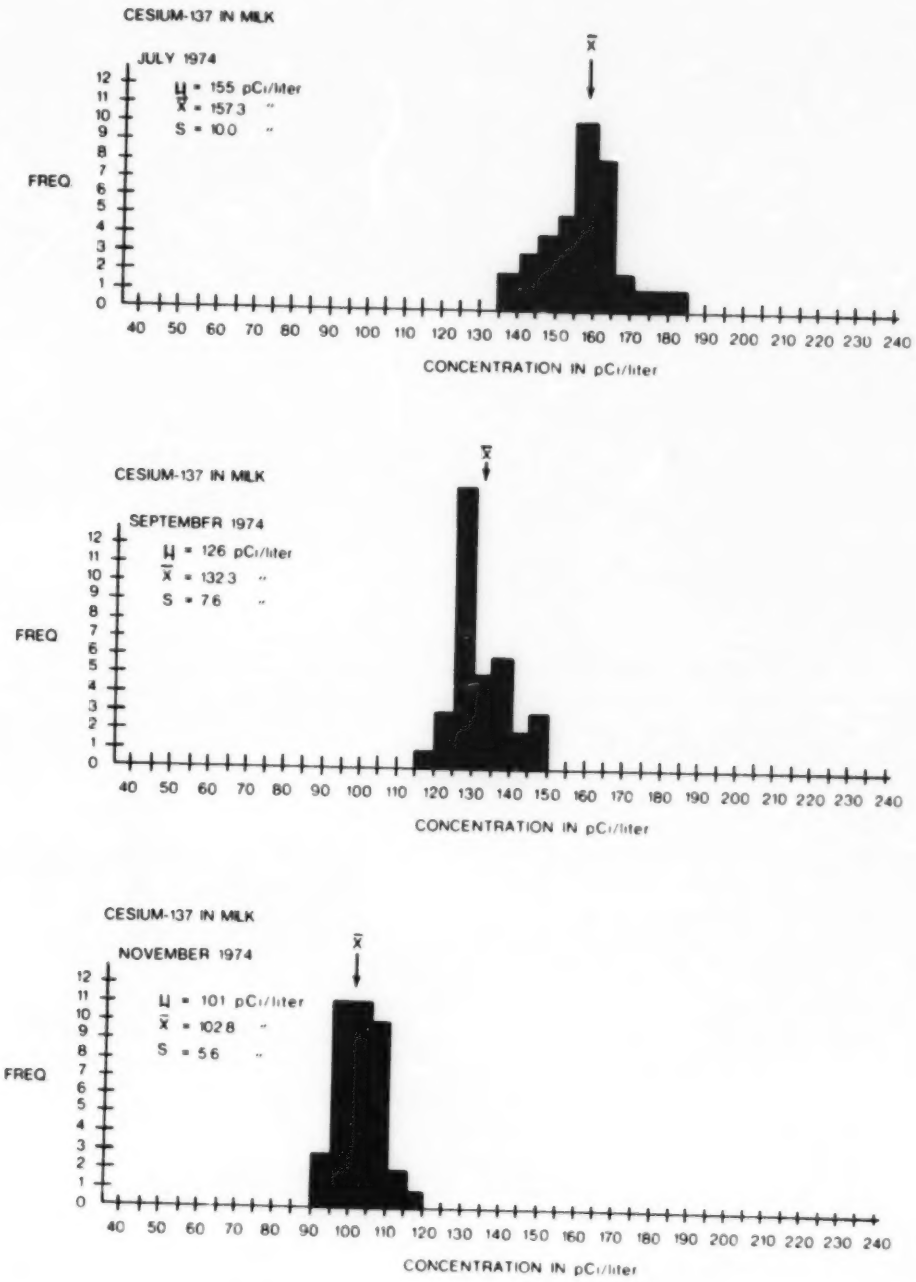


Figure 3-E. Histogram of Monthly Analysis for Cesium-137 (continued).

REFERENCES

1. F. Knowles, "Interlaboratory Study of Iodine-131, Cesium-137, Barium-140, Strontium-89, and Strontium-90 Measurements in Milk, May-July 1970," Technical Experiment 70-MKAQ-1, Analytical Quality Control Service, Bureau of Radiological Health (1970).

2. F. Knowles, "Rapid Field Method for Radiostrontium in Milk," Technical Report 70-TEC-1, Analytical Quality Control Service, Bureau of Radiological Health (1970).

APPENDIX. STATISTICAL CALCULATIONS

To illustrate the computations performed by the computer, example calculations are given using data for three actual samples analyzed at one laboratory (Laboratory D).

The experimental data are listed and the mean, range, and the experimental sigma are computed. These statistics provide measures of the central tendency and dispersion of the data.

The normalized range is computed by first finding the mean range, $\bar{R}$, the control limit, CL, and the standard error of the range, σ_R. The normalized range measures the dispersion of the data (precision) in such a form that control charts may be used. Control charts allow one to readily compare past analytical performance with present performance. In the example, the normalized range equals 0.3 $\bar{R}$ which falls inside the upper warning level, $\bar{R} + 2\sigma_R$. The precision of the results is acceptable.

The normalized deviation is calculated by computing the deviation and the standard error of the mean, σ_m. The normalized deviation allows one to readily measure central tendency (accuracy) through the use of control charts. Trends in analytical accuracy can be determined in this manner. For this example, the normalized deviation is -0.7 which falls within the upper and lower warning levels. The accuracy of the data is acceptable.

Finally, the experimental error of all laboratories, the grand average, and the normalized deviation from the grand average are calculated in order to ascertain the performance of all the laboratories as a group. Any bias in methodology or instrumentation may be found from these results.

EXAMPLE CALCULATIONS (Laboratory D Data)

Experimental data:

Known value = μ = 3273 pCi ^{3}H/liter urine on September 24, 1974

Expected laboratory precision = σ = 357 pCi/liter

Laboratory	Sample	Result
D	x_1	3060 pCi/liter
D	x_2	3060 pCi/liter
D	x_3	3240 pCi/liter

Mean = $\bar{x}$

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N} = \frac{9360}{3} = 3120 \text{ pCi/liter}$$

where N = number of results

Range = r

$$r = |\text{maximum result} - \text{minimum result}|$$

$$= |3240 - 3060| = 180 \text{ pCi/liter}$$

Experimental sigma = s

$$s = \sqrt{\frac{\sum_{i=1}^{N} (x_i)^2 - \frac{\left(\sum_{i=1}^{N} x_i\right)^2}{N}}{N - 1}}$$

$$= \sqrt{\frac{(3060)^2 + (3060)^2 + (3240)^2 - \frac{(3060 + 3060 + 3240)^2}{3}}{2}}$$

$$= 103.9 \text{ pCi/liter}$$

Normalized range = $w\bar{R} + x\sigma_R$

Mean range = $\bar{R}$

$$\bar{R} = d_2\sigma \qquad \text{where } d_2 = 1.693 \text{ for } N = 3^*$$

$$= (1.693)(357)$$

$$= 604.4 \text{ pCi/liter}$$

Control limit = CL

$$CL = \bar{R} + 3\sigma_R$$

$$= D_4\bar{R} \qquad \text{where } D_4 = 2.575 \text{ for } N = 3^*$$

$$= (2.575)(604.4)$$

$$= 1556 \text{ pCi/liter}$$

Standard error of the range = σ_R

$$\sigma_R = 1/3\ (\bar{R} + 3\sigma_R - \bar{R})$$

$$= 1/3\ (D_4\bar{R} - \bar{R})$$

$$= 1/3\ (1556 - 604.4)$$

$$= 317.2 \text{ pCi/liter}$$

$$w\bar{R} + x\sigma_R = 1\bar{R} + x\sigma_R$$

$$= 1\bar{R} + \left[\frac{r - w\bar{R}}{\sigma_R}\right]\sigma_R \qquad \text{for } r > \bar{R}$$

---

* Rosenstein, M., and A. S. Goldin, *Statistical Techniques for Quality Control of Environmental Radioassay*, AQCS Report Stat-1, U.S. Department of Health, Education and Welfare, PHS, November 1964.

$$w\bar{R} + x\sigma_R = w\bar{R} + o\sigma_R$$

$$= w\bar{R}$$

$$= \left[\frac{r}{\bar{R}}\right] \bar{R} \qquad \text{for } r \leq \bar{R}$$

$$= \left[\frac{180}{604.4}\right] \bar{R} \qquad \text{since } 180 < 604.4$$

$$= 0.30\ \bar{R}$$

Normalized deviation of the mean from the known value = ND

Deviation of mean from the known value = D

$$D = \bar{x} - \mu$$

$$= 3120 - 3273$$

$$= -153 \text{ pCi/liter}$$

Standard error of the mean = σ_m

$$\sigma_m = \frac{\sigma}{\sqrt{N}}$$

$$= \frac{357}{\sqrt{3}}$$

$$= 206.1 \text{ pCi/liter}$$

$$ND = \frac{D}{\sigma_m}$$

$$= \frac{-153}{206.1}$$

$$= -0.7$$

Experimental sigma (all laboratories) = s_t

$$s_t = \sqrt{\frac{\sum_{i=1}^{N} (x_i)^2 - \frac{\left(\sum_{i=1}^{N} x_i\right)^2}{N}}{N - 1}}$$

$$= \sqrt{\frac{162639133 - \frac{(49345)^2}{15}}{14}}$$

$$= 149 \text{ pCi/liter}$$

Grand average = GA

$$= \frac{\sum_{i=1}^{N} x_i}{N}$$

$$= \frac{49345}{15}$$

$$= 3290 \text{ pCi/liter}$$

Normalized deviation from the grand average = ND'

Deviation of the mean from the grand average = D'

$$D' = \bar{x} - GA$$

$$= 3120 - 3290$$

$$= - 170 \text{ pCi/liter}$$

$$ND' = \frac{D'}{\sigma_m}$$

$$= \frac{- 170}{206.1}$$

$$= - 0.8$$